



Application No: GB 9804949.7
Claims searched: 1-12

Examiner: Alan Kerry
Date of search: 14 July 1998

Patents Act 1977
Search Report under Section 17

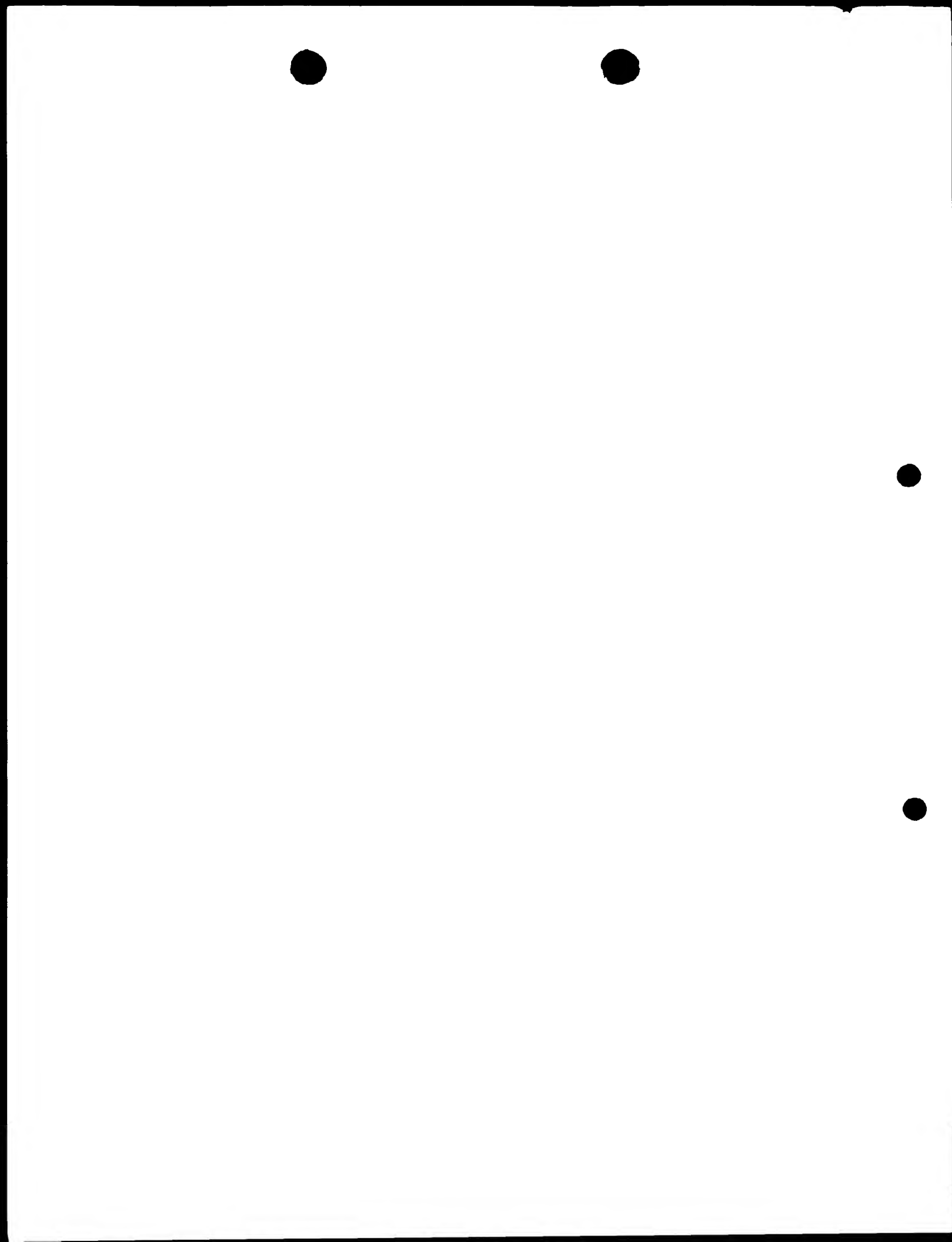
Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:
UK CI (Ed.P): C3V VAT, VEK
Int CI (Ed.6): C08L 83/04; C09D 7/12, 183/04; F16J 15/12
Other: Online databases: WPI, CLAIMS

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	GB 2122699 A (T&N) - see Claims 1-3 and Example 3	1 at least
A	GB 2093474 A (TEXON) - see whole document	1 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.





The
Patent
Office

REC'D 03 MAR 1999

WIPO PCT

PCT/GB 99/00491



INVESTOR IN PEOPLE

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP9 1RH

5

GB 99/00491

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

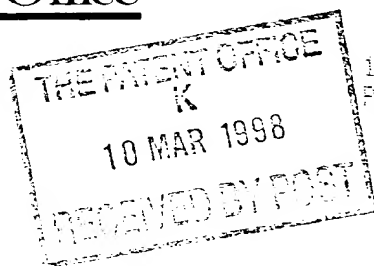
Dated

24.3.99



Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



10MAR98 E344124-1 001975
P01/7700 25.00 - 9804949.7

The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference

TNT 3011.GB

2. Patent application number
(To be filled in by the applicant)

10 MAR 1998

3. Full name, address and postcode of the or of each applicant (underline all surnames)

9804949.7

T&N TECHNOLOGY LIMITED
CAWSTON HOUSE
CAWSTON LANE
RUGBY, WARWICKSHIRE
CV22 7SA

Patents ADP number (if you know it)

755231001 ✓

If the applicant is a corporate body, give the country/state of its incorporation

ENGLAND

4. Title of the invention

GASKET COATING

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

P L DRURY, J A CRUX, G K GIBSON,
J HAMMERSLEY, J A MOFFAT
T&N PLC
BOWDON HOUSE
ASHBURTON ROAD WEST
TRAFFORD PARK
MANCHESTER
M17 1RA

Patents ADP number (if you know it)

5943790001 ✓

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number
(if you know it)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

YES

- a) any applicant named in part 3 is not an inventor, or
 - b) there is an inventor who is not named as an applicant, or
 - c) any named applicant is a corporate body.
- See note (d))

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form 0

Description 6

Claim(s) 2

Abstract 1

Drawing(s) 0

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

I/We request the grant of a patent on the basis of this application.

Signature

Date 2-3-98

DR A R BEGG, DIRECTOR, T&N TECHNOLOGY LIMITED

12. Name and daytime telephone number of person to contact in the United Kingdom

P L DRURY - 01788 528356

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

GASKET COATING

This invention is concerned with a gasket coating. In particular, the invention is concerned with a gasket coating for application to a surface of a gasket to provide improved sealing by filling small cracks and fissures in the surface of the gasket and/or of the member against which the gasket seals.

Gaskets are used for sealing between two members, eg two portions of the exhaust system of an internal combustion engine, and provide a seal around a passage which passes from one member to the other. Accordingly, a gasket has to be resilient in order to press against the members and provide a fluid-tight seal. In the case of an exhaust gasket, the seal is to prevent escape of exhaust gases. The resilience can be provided by utilising a relatively thick layer of resilient material such as a resilient rubber-based material but, where high temperatures (above 300°C) are experienced such as in an exhaust system, resilient materials would degrade and are not commonly used. Instead, the gasket comprises a sheet of metal, eg stainless steel, which is formed into resilient ridges (called "beads") which provide the seal. Thus, when such a gasket is clamped between two members, the clamping force compresses the beads which are resiliently deformed and press against the members along the lines of the beads. However, such gaskets normally do not provide as good a seal as is desirable because the beads are unable to enter into small cracks and fissures in the members so that gases and liquids can escape past the beads. It is known, in gaskets not utilised at such high temperatures as are experienced by exhaust gaskets, to provide metal beads with a thin coating (typically less than 200 microns in thickness) of a sealing-enhancing coating which will deform under clamping pressure to fill cracks and fissures. Known sealing enhancing coatings, however, degrade at higher temperatures so that they cannot

be used on, eg, exhaust gaskets for internal combustion engines.

It is an object of the present invention to provide a sealing enhancing coating which is suitable for use on a gasket or a portion thereof, eg bore seals, which will experience high temperatures in service.

The invention provides a sealing-enhancing coating for a gasket or a portion of a gasket, wherein the coating comprises flaky particles of chemically exfoliated vermiculite, at least 90% by weight of said particles having a thickness of no more than 30 microns, and no dimension greater than 1mm, the particles forming 10 to 90 wt% of the coating, the coating also comprising 50 to 10 wt% of an organic polymer binder which when formed into a film 1mm or less in thickness and heated to 300°C in free air for 24 hours either does not decompose or decomposes leaving a residue of at least 20% by weight of the film.

It is found that a coating according to the invention improves the sealing ability of gaskets which experience high temperatures in service, in particular gaskets with embossed sealing beads for example exhaust gaskets for internal combustion engines.

Exfoliated vermiculite is a known heat-resistant resilient material which provides both sealing and binding properties at high temperatures (eg above 300°C). Exfoliated vermiculite is conventionally formed by expanding mineral vermiculite using gas. Chemically-exfoliated vermiculite (CEV) is a form of exfoliated vermiculite which is formed by treating the ore and swelling it in water. In one possible preparation method, the ore is treated with saturated sodium chloride solution to exchange magnesium ions for sodium ions, and then with n-butyl ammonium chloride to replace sodium ions with n-

$C_4H_9NH_3$ ions. On washing with water swelling takes place. The swollen material is then subjected to high shear to produce an aqueous suspension of very fine (diameter often below 50 microns) vermiculite particles.

It is known to utilise chemically-exfoliated vermiculite in a sealing layer, ie the layer which provides the sealing force by compression thereof, of a sheet gasket, eg an automotive head gasket. For example, GB 2 123 034 B describes making a flexible sheet material, eg for a gasket, by subjecting an aqueous suspension to electrophoresis. The suspension contains an expanded layer silicate, eg CEV with a particle size below 50 microns, and a dispersed organic polymeric material, eg acrylic polymer, acrylonitrile-butadiene copolymer, epoxy resin, or natural rubber. However, these flexible sheet materials are not suitable for use as a sealing-enhancing coating on an exhaust gasket because the polymeric material would degrade at high temperatures and the coating would become less effective.

In a coating according to the invention, the polymer binder is preferably a silicon-containing polymer, eg a silicone resin or a siliconate. Also possible are PTFE, phenolics, and fluoroelastomers.

Preferably, a coating according to the invention also comprises a solid lubricant eg particles of graphite, molybdenum disulphide, hexagonal boron nitride, calcium difluoride, or PTFE (PTFE also serving as a binder).

A coating according to the invention may also comprise a flaky filler, eg mica, milled thermally exfoliated vermiculite, or aluminium flake.

The coating also, preferably, comprises a supplementary inorganic binder/adhesion promoter, eg a

water-soluble alkali silicate, especially lithium silicate.

Preferably, a coating according to the invention also comprises a waterproofing agent acting on at least one of the chemically exfoliated vermiculite and supplementary inorganic binders.

Preferably, none of the ingredients of a coating according to the invention should prematurely interact with one another. In particular, none of the ingredients should flocculate the CEV or precipitate the alkali silicate.

The invention also provides a gasket comprising a metal sheet having thereon elongated embossed beads, the beads having thereon a coating according to the invention, wherein the coating has a thickness of less than 200 microns.

There now follows a detailed description of two examples of sealing-enhancing coatings which are illustrative of the invention.

In order to form the first illustrative coating, the following were mixed together:

- (a) 50g of chemically-exfoliated vermiculite suspension (15% solids in water). This was obtained from W R Grace & Co under the designation Microlite HTS. The particles of CEV were flaky and had a size distribution such that at least 90% by weight had a thickness of no more than 30 microns and no dimension greater than 1mm. Specifically, the vermiculite particles have less than 33% above 45 microns diameter and an aspect ratio of at least 100.

- (b) 5g of graphite flake particles (grade Hart 400 from Colin Hart Minerals). This graphite is milled so that 96% of the particles pass through a 37 micron sieve. These particles were included to provide a solid lubricant.
- (c) 5g of aluminium powder (grade Fine 124 from Ronald Britton & Co). The mean particle size of the aluminium is 16-24 microns with less than 1% being retained on a 160 micron sieve. This powder formed a water-proofing agent acting on the inorganic binder(s).
- (d) 5g of mica particles (SX 300 from Microfine Minerals). 1-4% of the mica particles are retained by a 20 micron sieve. The mica was included as an inorganic filler.
- (e) 10g of methyl phenyl silicone resin emulsion (MP 42E from Wacker). This was a suspension in water at 42% solids and provided an organic polymer binder. This resin has an ignition point of 465°C. This binder, when formed into a film 1mm or less in thickness and heated to 300°C in free air for 24 hours decomposes leaving a residue of at least 20% by weight of the film, the residue being silica which has some binding properties.
- (f) 5g of lithium silicate solution (23% solids). This was obtained from Crossfield under designation L40 and provided an inorganic binder in addition to the organic binder provided by the silicone resin and the CEV. The lithium silicate takes over some of the binding function at higher temperatures (at which the silicone resin decomposes). The lithium silicate may also promote bonding to steel substrates.

The mixture was sprayed onto the surface of an embossed stainless steel sheet of a gasket. The embossments formed sealing beads which were covered by the sprayed mixture. The mixture was then dried forming a coating over the beads having a thickness of less than 200 microns.

In order to form the second illustrative coating, the following were mixed together:

- (a) 200g of chemically-exfoliated vermiculite particles as mentioned at (a) of the first example.
- (b) 20g of graphite flake particles as mentioned at (b) of the first example.
- (c) 40g of silicone resin as mentioned at (e) of the first example.
- (d) 20g of water.

The mixtures of the second example was sprayed on to the surface of an embossed stainless steel gasket sheet. The mixtures were then dried to form a coating less than 200 microns in thickness.

CLAIMS

- 1 A sealing-enhancing coating for a gasket or a portion of a gasket, wherein the coating comprises flaky particles of chemically exfoliated vermiculite, at least 90% by weight of said particles having a thickness of no more than 30 microns, and no dimension greater than 1mm, the particles forming 10 to 90 wt% of the coating, the coating also comprising 50 to 10 wt% of an organic polymer binder which when formed into a film 1mm or less in thickness and heated to 300°C in free air for 24 hours either does not decompose or decomposes leaving a residue of at least 20% by weight of the film.
- 2 A coating according to claim 1, wherein the polymer binder is a silicon-containing polymer.
- 3 A coating according to claim 2, wherein the polymer binder is a silicone.
- 4 A coating according to claim 2, wherein the polymer binder is a silicate.
- 5 A coating according to claim 1, wherein the polymer binder is selected from PTFE, phenolics, and fluoroelastomers.
- 6 A coating according to any one of claims 1 to 5, wherein the coating also comprises particles of a solid lubricant.
- 7 A coating according to any one of claims 1 to 6, wherein the coating also comprises a flaky filler.

- 8 A coating according to any one of claims 1 to 7, wherein the coating also comprises a supplementary inorganic binder.
- 9 A coating according to claim 8, wherein the supplementary inorganic binder is lithium silicate.
- 10 A coating according to any one of claims 1 to 4, wherein the coating also comprises a waterproofing agent acting on at least one of the chemically exfoliated vermiculite and the supplementary inorganic binder.
- 11 A coating substantially as hereinbefore described with reference to the illustrative examples.
- 12 A gasket comprising a metal sheet having thereon elongated embossed beads, the beads having thereon a coating according to any one of claims 1 to 10, wherein the coating has a thickness of less than 200 microns.

ABSTRACT

Gasket Coating

A sealing-enhancing coating for a gasket or a portion of a gasket comprises flaky particles of chemically exfoliated vermiculite, at least 90% by weight of said particles having a thickness of no more than 30 microns, and no dimension greater than 1mm. The particles form 10 to 90 wt% of the coating. The coating also comprises 50 to 10 wt% of an organic polymer binder which when formed into a film 1mm or less in thickness and heated to 300°C in free air for 24 hours either does not decompose or decomposes leaving a residue of at least 20% by weight of the film.

... PCT/GB 09/0029,
... 15.2.90.

T-N Limited.